

A Vacuum Ultraviolet Spectrophotometric System**James F. Spann¹, Charles E. Keffer², Muamer Zukic²**¹NASA/Marshall Space Flight Center

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Abstract

The development of a vacuum ultraviolet spectrophotometric system for measuring transmittance and reflectance at variable angles is presented. Using various detectors and sources, the spectrophotometric system has been used for wavelengths from 80 nm to 300 nm with optical components up to 80 mm in diameter. The capability exists to make measurements through the visible range.

Key Words: Spectrophotometer, ultraviolet filters, transmittance, reflectance

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A. Introduction

The need for spectrophotometric measurements of reflectance and transmittance in the vacuum ultraviolet (VUV) region of the spectrum, from 0.2 nm to 200 nm,¹ has become more important in recent years with the development of all-dielectric narrowband filters in the far ultraviolet (FUV) from 200 nm to 100 nm² and the extreme ultraviolet (EUV) from 100 nm to 30 nm,³ and multilayer normal incidence mirrors in the x-ray ultraviolet (XUV) from 30 nm to 0.2 nm.⁴ The characterization of optical materials and measurements of spectral performance of optical components has been an essential ingredient in the recent developments.

Spectrophotometric systems that have been described in the literature are typically for in-air measurements⁵⁻⁸. However, the use of many optical components render these techniques impractical for the VUV region. There have been some references to techniques which can be employed in the VUV region,⁹ and a commercial system is available for small samples up to 50 mm square.¹⁰ Some work has been done related to developing a method of calibrating VUV radiation sources.¹¹ However, the accuracy of spectrophotometric measurements is derived from system design and characterization of all sources of error and not from a fundamental physical standard.

In this paper, we describe a spectrophotometric system we have developed which is useful in the VUV region of the spectrum. The system has been used for various activities including the characterization of optical properties of thin films^{12,13} and space flight hardware for the Ultraviolet Imager (UVI) of the Global Geospace Science (GGS) POLAR spacecraft.¹⁴

B. Measurement System

VUV optics are extremely sensitive to contamination, especially hydrocarbon deposition. In addition, multilayer dielectric optics cannot be easily cleaned without damage to the coating. Scattered light due to particulate contamination is also a serious problem in the VUV range. In order to minimize exposure of test optics to these types of contamination, the spectrophotometric system is maintained in a class 1000 clean area in a stainless steel vacuum chamber. Carbon vane blowers or venturi pumps are used in the rough vacuum range and cryogenic pumps are used in the high vacuum range in order to avoid contamination from hydrocarbon-based pump fluids. The vacuum system operates with a base pressure in the 10^{-7} torr range.

For FUV measurements, a high pressure arc discharge deuterium lamp is used as the source. A 0.2 m vacuum monochromator, with a concave holographic grating, coupled to a 1.5 m focal length collimating UV enhanced mirror system produces a monochromatic collimated incident beam. The beam can be directed via a two axis gimbal system to which the collimating mirror is mounted. The beam is apertured to ensure that the entire beam is incident on the detector. Figure 1 schematically illustrates the system. The detector is a photomultiplier tube (PMT). Depending on the wavelength range of interest, one of four PMTs referenced in Table 1 is used.

The PMT with a bialkali photocathode can be configured to have an additional Pyrex window which is coated with a layer of sodium salicylate. The sodium

salicylate layer provides a wavelength shift from the VUV to 420 nm.¹⁵ Care must be exercised when using sodium salicylate due to its fluorescent nature and propensity to powder when physically disturbed. Figure 2 shows the configuration for this detector. The layer of sodium salicylate was deposited by using an atomizer to spray a suspension of ethanol and sodium salicylate onto a Pyrex substrate. The thickness was determined by weighing the substrate before deposition and after the layer dried. A layer with an average thickness of 1.2 mg cm^{-2} was deposited. The bialkali PMT was chosen because its peak response at 420 nm matched the fluorescent emission peak of sodium salicylate.

The optical samples to be measured are housed in a filter wheel. For 25.4 mm and 12.7 mm diameter samples, an eight-position filter wheel is used. This allows up to six samples to be measured with one reference position and one background position. For larger samples or odd-shaped samples, another filter wheel is used with four positions. Either filter wheel is mounted on a platform that can rotate about an axis that is coincident with the front surface of the sample. The PMT is mounted on an arm which is rotated independently about the same axis (see Figure 1). Reflectance and transmittance measurements can be made for multiple angles of incidence. The spectral characteristics of the incident beam are determined by the wavelength setting of the monochromator and the width of the variable-width slits. Two different gratings are used in the monochromator. An iridium-coated grating is used for measurements from 30 nm to 120 nm. An Al/MgF₂ coated grating is used for the measurements from 120 nm to 500 nm. Spectral resolution to 0.1 nm is achievable.

The filter wheel position and monochromator wavelength setting are controlled using stepper motors controlled by a PC. The PMT signal is acquired using an

IEEE 488-controlled picoammeter. Integrated software allows automated spectrophotometric measurements to be made over the desired wavelength range.

For measurements that require larger signal and do not require a collimated incident beam, the system can be moved into another chamber schematically depicted in Figure 3. The significant difference in this configuration is the lack of collimating optics. The divergence of the incident beam is on the order of 10 mrad, defined by the f/4.5 monochromator optical system. For the measurements made to date, this has not been a factor. This configuration is better suited for EUV measurements because of fewer reflections. The source used for EUV measurements is a capillary glow dc discharge lamp.¹⁶

C. Measurement Process

To measure the transmittance of a sample the filter wheel is oriented to the desired angle of incidence and the PMT is positioned in line with the incident beam. The beam is apertured down so that it will fall entirely on the detector active area even if there is a translation of the transmitted beam due to the sample substrate thickness. The monochromator is set to the desired spectral resolution and initial wavelength. The filter wheel is rotated to the reference position where the incident beam passes unattenuated through to the detector. In this position, the reference flux is measured. The filter wheel is then rotated to the sample position and the signal measurement is made. If there are other samples, then a signal measurement is made for each one as the filter wheel is rotated to each of those positions. The filter wheel is returned to the reference

position for a second reference measurement. At some point a background measurement is made by rotating the filter wheel to the position where the incident beam is blocked. This completes the measurement cycle for that wavelength. The incident beam's wavelength is changed and the cycle is repeated until measurements at all wavelengths are completed. The effects of source intensity drift are minimized using this sequence of measurements; however, the effects of non-repeatability of the filter wheel positioning are enhanced. Alternatively, the entire wavelength range is scanned at each filter wheel position beginning with the first reference scan. A signal scan for all wavelengths for the first sample is made before going to the next sample; a background measurement is taken; and another reference scan is completed. This measurement sequence minimizes the effects of filter wheel position repeatability, but is more sensitive to nonlinear drifts of the source intensity.

For reflectance measurements, the same steps are taken for either of the measurement sequences with two exceptions. The detector is positioned so that the specular reflection of the beam off a calibrated reference mirror falls entirely on the active area of the PMT. The calibrated reference mirror is used to calculate the unattenuated reference beam flux by dividing the flux measurement by the known reflectance of the mirror. The background measurement is made by rotating the filter wheel to the open position which corresponds to the reference position for a transmittance measurement.

For each spectrophotometric measurement, a control sample is measured along with the sample(s). One control sample is used for all transmittance measurements and one for all reflectance measurements. This enables a quick evaluation to be done on the validity of a particular measurement, and provides a

measure of repeatability and an indication of system contamination. It also provides a measure of continuance if the spectrophotometric system is moved from one vacuum chamber to the other.

D. Error Analysis

The spectrophotometric measurements are made by taking the ratio of the reflected or transmitted beam flux I_s , called signal, and the unattenuated beam flux I_o , called reference. The measured reflectance R or transmittance T is

$$R = \frac{I_s}{I_o} \quad (1)$$

$$T = \frac{I_s}{I_o} \quad (2)$$

Because both of these measurements are made using the same source and detector, errors resulting from cross calibration of sources and detectors are eliminated. Errors due to source output and detector response drift are minimized by measuring the reference before and after the signal. The reference is linearly extrapolated to the time the signal was measured. In addition, a background measurement I_b is made with the incident beam blocked. For transmittance measurements

$$T = \frac{I_s - I_b}{I_o(t_s) - I_b} \quad (3)$$

where t_s denotes the time the signal was measured.

A calibrated mirror is used for reflectance measurements. The reference is determined by the measured signal reflected off the calibrated mirror I_m divided by the known reflectivity of the mirror R_m , or

$$I_o = \frac{I_m}{R_m}. \quad (4)$$

For reflectance measurements

$$R = \frac{(I_s - I_b)R_m}{I_m(t_s) - I_b}. \quad (5)$$

The fractional errors associated with the source and detector drift are small (<0.003) over the time interval between measurements which is typically less than 1 minute. The background measurement is 2 to 3 orders of magnitude smaller than the signal. The noise is due to thermal noise of the detector. The signal and reference measurement uncertainty is a combination of the source and detector noise. The fractional error in R or T for a given measurement is typically less than 0.015. The absolute value of R or T is determined by accessing the repeatability of a measurement. Figure 4 is a plot of the averaged reflectance measurements of a control sample used for the much of the reflectance measurements done to date. A total of 150 measurements are included in this average over a period of 31 months. Measurements in both vacuum chambers are included. The error bars represent the repeatability that is achieved. For the peak, the absolute fractional repeatability is less than 0.04. For values around $R=0.05$, the absolute fractional repeatability is 0.17.

E. Conclusion

By using various detectors and sources with a single reflection monochromator in a hydrocarbon free vacuum system, VUV spectrophotometric measurements have been made to characterize thin films and multilayer optical components. The absolute fractional repeatability of measurement has been demonstrated to be better than 0.003. The capability exists to measure the reflectance and transmittance of different-sized samples, at multiple angles of incidence, and over the VUV and into the visible spectral range.

With advances of optical components in this wavelength range, requirements for an improved capability will be forthcoming. Known improvements to the current system that can be made include the incorporation of photon counting or lock-in amplifier techniques for weak signals, for example, when attempting to accurately measure small out-of-band filter reflectance or transmittance values.

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Photocathode	Window	Spectral Response	
		Range (nm)	Peak (nm)
CsI	MgF2	115 ~ 200	140
CsTe	MgF2	115 ~ 320	210
Bialkali	Quartz	160 ~ 650	420
Bialkali	Quartz with added sodium salicylate coated Pyrex window	20 ~ 350	150

Table 1. PMT photocathode and window materials and spectral response characteristics.

Figure Captions

Figure 1. Schematic of collimated spectrophotometric configuration.

Figure 2. Configuration of the bialkali PMT with a sodium salicylate coated window.

Figure 3. Schematic of uncollimated spectrophotometric configuration.

Figure 4. Reflectance measurement for RF135 control sample. The values are averages of 150 runs made over a period of 31 months. The error bars represent one standard deviation.

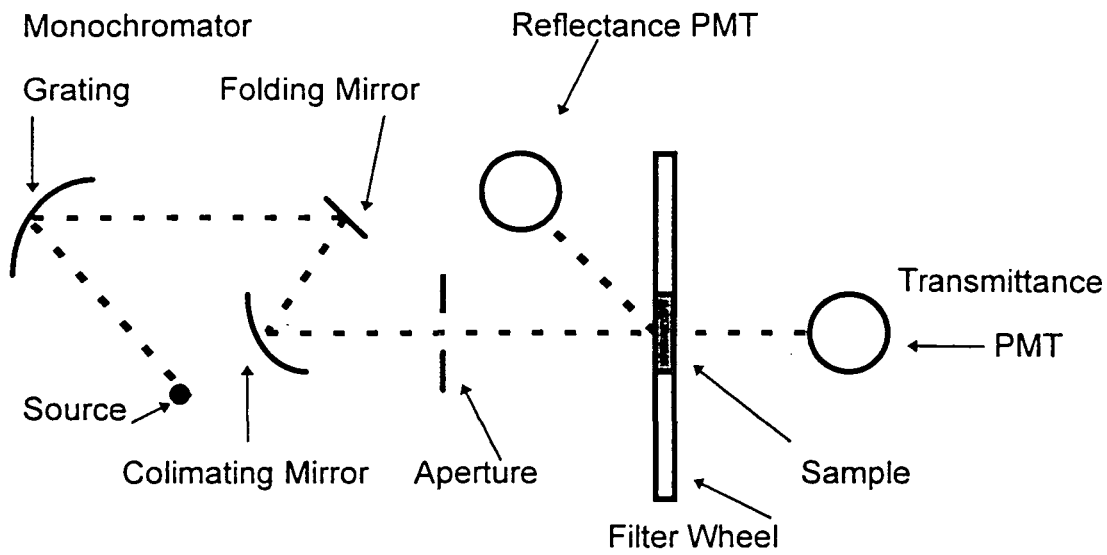


Figure 1. Schematic of collimated spectrophotometric configuration.

Figure 1

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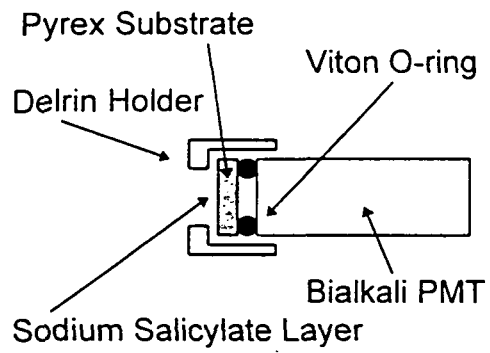


Figure 2. Configuration of the bialkali PMT with a sodium salicylate coated window.

Figure 2

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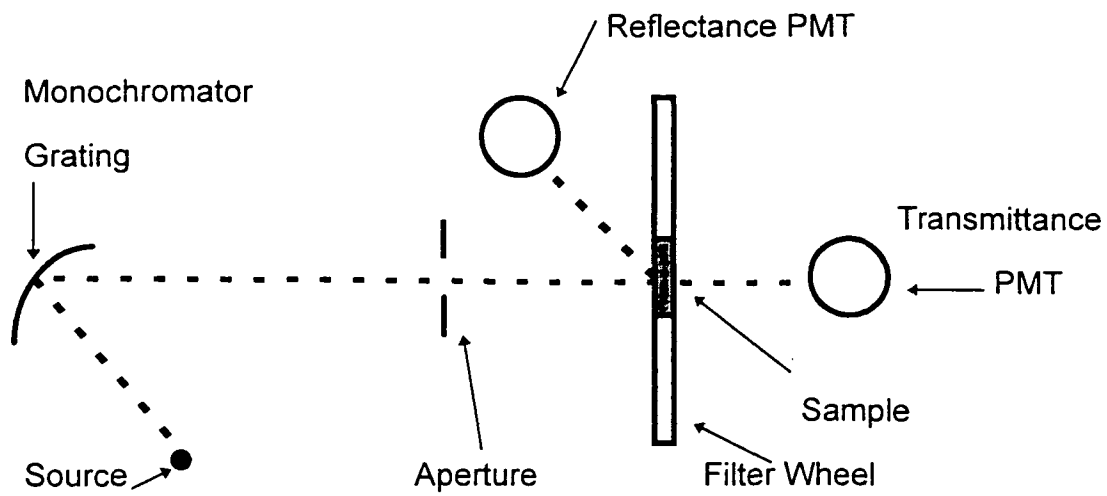


Figure 3. Schematic of uncollimated spectrophotometric configuration.

Figure 3

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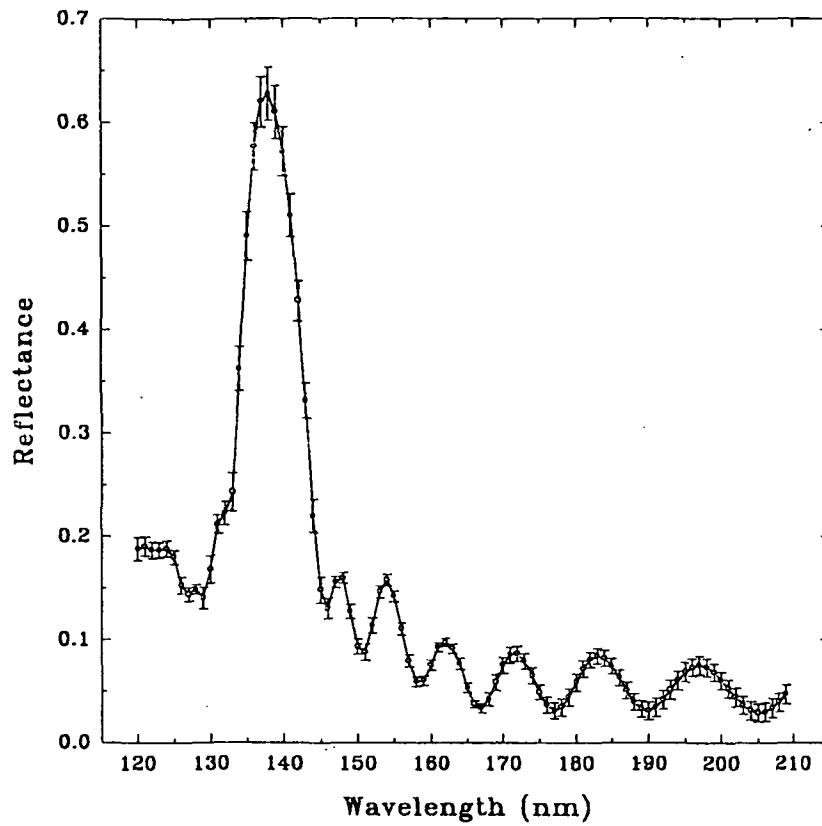


Figure 4. Reflectance measurement for RF135 control sample. The values are averages of 150 runs made over a period of 31 months. The error bars represent one standard deviation.

Figure 4

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